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Preliminary Hazard Analysis of Supercritical Fluid Separation of Energetic Materials

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Preliminary Hazard Analysis of Supercritical Fluid Separation of Energetic Materials

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Abstract

The Department of Defense (DOD) has a large inventory of ammunition, some of which is becoming older than desired for retention in storage. More than 400 kton are currently scheduled for demilitarization. Of this material, a significant fraction is Composition B, which is 60% RDX and 40% TNT, with 1% wax added. Research at the U.S. Army Research Laboratory (ARL) and elsewhere, particularly at the Phasex Corporation, Lawrence, MA, has demonstrated the feasibility of separating the energetic moieties by use of supercritical CO₂. No special hazards were identified, and no chemical incompatibilities were found. The system is environmentally friendly in that it is a closed-loop system, and no hazardous materials can escape. Also, this system is designed for remote operation. This report deals with potential hazards from the scale-up of the system with which the feasibility of the concept was demonstrated.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the frequent and detailed discussions of this subject with Dr. Jeffrey Morris, U.S. Army Research Laboratory, and J. W. Wright and R. S. Cohen, Applitech Corporation, in the refinement of the system being developed. The authors also thank James E. Cocchiaro of the Chemical Propulsion Information Agency, The Johns Hopkins University, Columbia, MD, for his help in finding and providing reference materials useful to this research. Funding for this task was provided by the Strategic Environmental Research and Development Program (SERDP).

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1. PURPOSE

The objective of this study was to do a preliminary risk assessment on a design for the separation of the explosive cyclotrimethylenetrinitramine (RDX) from the explosive formulation known as Composition B (Comp B) (60% RDX/40% trinitrotoluene [TNT] with 1% wax added) by means of a supercritical-fluid (SCF), carbon dioxide (CO₂), processing.

David J. Van Horn [1, chap. 4] defines a Preliminary Hazard Assessment (PHA) as follows: "A PHA has traditionally taken the form of inventorying all the materials and equipment to be used and deciding what are the hazardous elements. Intuition, experience and judgement are applied to determine what can lead to accidents and whether the risk is acceptable or the hazard must be corrected by controls and/or contingency plans..." Our approach takes this process considerably further. Not only are the materials and equipment considered, but also the dynamics of the entire process.

2. EMPHASIS

The considerations that are raised here are based on Army safe methods and practices as found in AMC-R 385-100 [2] and Quantity-Distance Tables [3]; Navy and Air Force regulations are similar [4,5].

The interaction of energetic materials with supercritical CO₂ and the various elements of the system, as these materials proceed through the separation process, was emphasized.

Energetic materials (Comp B) react in response to various stimuli: elevated temperature, electrical potential, friction/squeezing, sparks, shock, or impact. Combinations of stimuli are important (e.g., pressure with elevated temperature). Particular attention has been given to these parameters as the Comp B is followed through the proposed system as the separation process is carried out.

An attempt was made to anticipate problems and suggest contingency plans. During routine operation of the system, certain sets of conditions may arise that could cause malfunctions (e.g., a sudden drop in SCF pressure). In such a situation, the operator must be able to take immediate and effective action.

3. BACKGROUND

The Department of Defense (DOD) has a large inventory of ammunition, some of which is becoming older than desired for retention in storage. More than 400 ktons are scheduled for demilitarization [6]. A significant percentage of this material is Comp B. Some preliminary work on this process has been performed by Morris [6], in which he was able to demonstrate the successful extraction of TNT from Comp B. Earlier work with SCF was done for the U.S. Army Research Laboratory (ARL) by Phasex Corporation, Lawrence, MA [7, 8]. All of this work was done through the use of supercritical CO₂. No special hazards were identified, and no chemical incompatibilities were found. The system is environmentally friendly in that it is a closed-loop system, and no hazardous materials can escape. Also, this system is designed for remote operation. In consideration of scale-up of this process, a Strategic Environmental Research and Development Program (SERDP) task was defined.

The explosives to be separated by this process would have been removed from all hardware before being treated with SCFs. The process involves the selective solubilization of TNT from Comp B, followed by precipitation of the TNT from this solution when the pressure is reduced. This is a batch process. When the TNT has been extracted, it is removed in its molten form from the system, the extractor is depressurized, and the RDX is removed.

4. EXCLUDED HAZARDS

The previous statement excludes a number of risks: poison(ing); bursting/leaking pipes, fittings, and equipment; non-high-explosive (HE) related burning/scalding of personnel and equipment; falling equipment; spills; high-temperature/pressure liquid or air; asphyxiation; and

any hazard not associated with the presence of Comp B in the system. Transportation of Comp B to and away from the system was also not considered; however, its entry and removal from the system was.

5. PROCEDURE

Each step in the process was analyzed to determine if the stimuli to which Comp B will respond were present, and in what degree. To avoid gaps in this survey, the Comp B was followed from its introduction into the system, through the system, to the departure of its moieties from the system. It was assumed that the Comp B had been manufactured in accordance with the appropriate Mil. Spec., and that it had not become contaminated before being submitted to this separation process.

6. DESCRIPTION OF SCF EXTRACTION SYSTEM

Figure 1 is a schematic diagram of the current version (August 1996) of the ARL SCF-extraction system. The heart of this system is a 125-l extraction vessel that operates at 6,000 psi and 100 °C. It uses a closed-loop flow of CO₂—gas-to-liquid-to-supercritical, back to the gaseous state, and then back around the loop. These phase transitions are detailed in Figures 2 and 3.*

The closure mechanism shown in Figure 1 is a breech-lock closure, operated by the pneumatic-hydraulic system depicted in the lower left of the figure. In the next section of this report, there is a recommendation concerning this type of mechanical activator (viz., to use only hydraulics, and avoid pneumatics for this operation). The cover-lift function was not indicated in the figure; however, it will likely be identical to the final version of the cover piston slide actuators.

* Figures 2 and 3 were provided by Mssrs. George Nauflet and Robert Farncomb of the Naval Surface Warfare Center, Naval Ordnance Station, Indian Head, MD. The data depicted in the figures were ascribed to Dean [9].

The makeup CO₂ feed enters the system at the lower right of the figure. The pump supplies the pressure required (critical pressure is 1070.4 psi), and the preheater raises the temperature to a point above the CO₂ critical temperature of 31.1° C. Normal operating conditions for the RDX extraction vessel are a pressure of 6000 psi and a temperature of 100 °C, well into the supercritical region of the phase diagram of Figure 2. Supercritical CO₂ selectively dissolves the TNT from the Comp B, leaving the RDX in the extraction vessel. The dissolved TNT is carried by the CO₂ through various valves and filters to the pressure-control valve, physically located at the entrance to the first separator, where the pressure is drastically reduced. Under these conditions, the CO₂ is no longer supercritical, and the TNT drops out of solution, but it is still liquid and, thus, can easily be drained off as indicated, as one of the end products of this separation process. The other end product, RDX, must be removed from the extraction vessel after its pressure is reduced to ambient and the breech-lock closure opened.

The previous description of system operation is very brief and leaves out many of the functional details, but it will suffice for this hazard analysis. Section 7 constitutes the heart of this report.

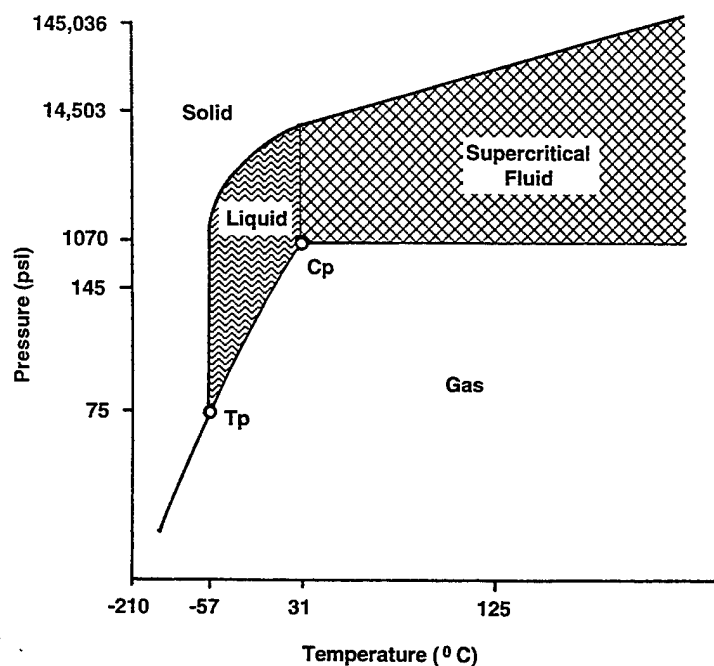


Figure 2. Carbon dioxide phase diagram.

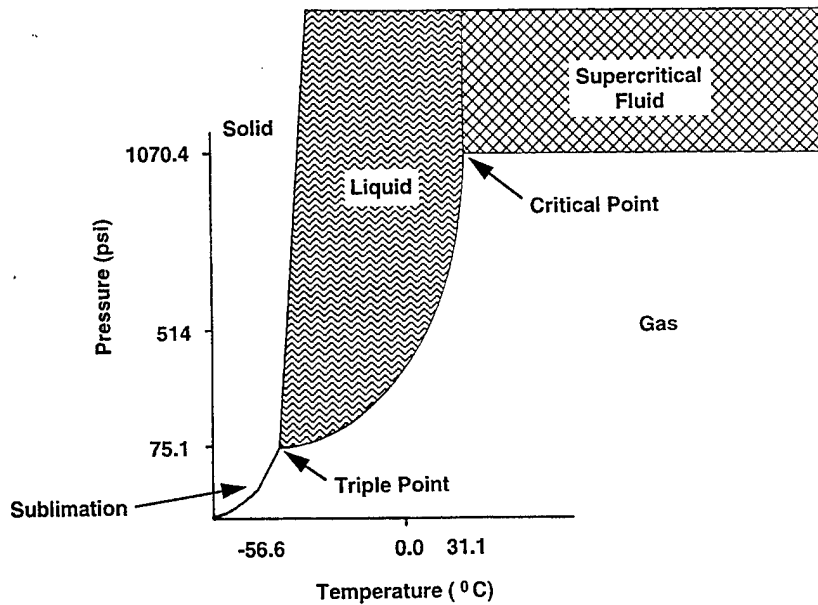


Figure 3. Detail of carbon dioxide phase diagram.

7. SAFETY CONSIDERATIONS AND RECOMMENDATIONS

(1) Filters will require monitoring so that the operation can be halted before a filter becomes so obstructed as to cause a problem in the functioning of the system. Remotely monitored pressure difference transducers should be installed across the filters that have a greater probability of clogging. These transducers should be monitored automatically as part of the computer program that controls the system operation. An annunciator should warn the operator of the clogging situation, and the filters should be isolated by valves so that they can be opened and cleaned safely while the system is in operation.

(2) Means must be built into the system to allow filters to be opened and cleaned safely. If the operation is to continue while this is being done, filters with the necessary valving must be installed parallel to these filters.

(3) The operating system should be electrically isolated from the control system. Optical isolation of the transducers from the readout instrumentation is recommended.

- (4) Potential voltage surges can be limited by zener diodes with associated circuitry.
- (5) Motors should be rated as Groups C and D (i.e., nonsparking for a dusty environment).
- (6) Hydraulic cylinders should have manual lockout valves to prevent them from operating at the wrong time.
- (7) Static electricity must be controlled. Single-point grounding is recommended. Ground loops should be avoided where possible.
- (8) Lightning protection for the area in which the system is to be operated is recommended.
- (9) Faraday shielding of the area containing the extraction vessel may be advisable. It may be possible for a "blast blanket" to serve a double purpose: to act as a Faraday shield as well as to contain an explosion.
- (10) The fluids, containing energetic materials, flowing through this system will generate static electricity. To avoid an internal discharge, it may be wise to install transverse, conductive, grounded screens. A coarse mesh would likely be acceptable. Such screens would also create turbulence, promoting discharge to the walls of pipes and containers.
- (11) Most RDX contains some cyclotetramethylenetetranitramine (HMX) that is usually in the β isomorph. Sensitivities of the four HMX isomorphs are in the sequence: $\delta > \gamma > \alpha > \beta$, the β isomorph being the least sensitive. Good temperature control is necessary to prevent a morphology change, leading to greater sensitivity.
- (12) A way to greatly reduce the load on the extraction vessel is to prefilter the RDX from the molten Comp B. The separated TNT would be relatively free of RDX, and the RDX would have only a small amount of TNT still associated with it, which the SCF system could easily remove.

(13) The recovered RDX must be wet down with 30 wt-% of a 60/40 alcohol/water mixture to ensure safety in storage and shipment.

(14) A Standing Operating Procedure (SOP) is required for this operation.

(15) A filter bag should be placed inside the mesh basket to facilitate removal of the RDX/HMX.

(16) Replace the combination pneumatic/hydraulic systems with straight hydraulic systems. Hydraulic fluid is almost incompressible and, thus, control can be much more precise.

(17) Preheat the SCF as it enters the extraction vessel to ensure the super critical condition of the fluid. Use an explosion-proof heater (e.g., a hot-liquid heat exchanger).

(18) Electrical potential must be well insulated from energetic materials at transducers and readouts. Optical links could be used to advantage in these applications.

(19) To stop fragments from an exploding extraction vessel, an outer vessel or shield surrounding the extraction vessel is recommended. The spacing between the extraction vessel and this shield must be sufficiently large so as to allow fragmentation of the inner vessel before striking the shield. This outer "vessel" should be well vented to the atmosphere during operation. Further containment can be accomplished by use of a blast blanket to catch spall and fragments. Refer to item 9.

(20) RDX crystals have many voids and inclusions. If shocked, these voids become initiation sources for further reaction, often leading to explosion. In pressurizing Comp B (RDX) with SCF CO₂, the rate of pressure increase should be low so as to avoid activating these voids. One must avoid RDX crystal fracture. However, the liquid CO₂ will act as a heat sink, minimizing this risk.

- (21) Use an oxygen sensor in the pit and all low areas to avoid asphyxiation.
- (22) The pneumatically operated valves shown do not have a positive closure. Air pressure opens them, and a spring returns them closed on all valves indicated in Figure 1 by open bowties. This may pose a problem.
- (23) Place the inlets to the separators in the immediate vicinity of the pressure letdown so that significant condensation will not occur upstream of the separators.
- (24) There must be no pathway for HE to go between the inside walls in the extraction vessel.
- (25) Avoid internal screw threads. If threads must be used, make them external. Better options are the pin closure and the breech-lock closure.
- (26) Avoid sparking metal to sparking metal contact for movable parts anywhere in the vicinity of HE.
- (27) Evacuate the system before filling with CO₂, to avoid the presence of oxygen in the system. Having an oxidizing agent in the system with the HE can be hazardous as related by Douglas Raynie [10] with respect to the use of nitrous oxide in SCF extractions.
- (28) The hazard radius should be calculated from the mass of the HE and the extraction vessel. It may be possible to use higher loadings than planned for a greater throughput rate.
- (29) Vapor-pressure and gas-flow calculations indicate a TNT vapor loss rate of about 1.4 g/hr into the system at a temperature of 85° C. At 30° C, the approximate temperature of the fluid as it enters the carbon filter just before being recycled, the TNT vapor pressure is lower by about a factor of 10³. The use of a chiller or trap before the carbon filter to trap out TNT condensate would significantly increase the useful life of the filter.

(30) A plan for the generation of environmental impact statements must be made for the planned sites of operation, including a spill plan for solids, liquids, and vapors.

(31) Since this system is intended to treat energetic materials belonging to all Services in the DOD, a joint service agreement on standards should be established.

(32) The provisions that ensure safety must be properly protected and frequently inspected for functional validity.

8. LESSONS LEARNED FROM VISIT TO WESTERN AREA DEMILITARIZATION FACILITY (WADF)

The environment, equipment, and safety precautions at WADF are relevant to the handling of explosives in this task.

A visit was made to this facility at Hawthorne, Nevada, in May 1996, for the purpose of observing a demilitarization operation. WADF currently processes approximately 12,000 short tons of obsolete, out-of-date, or unserviceable conventional ammunition per year. This is about half of their current capacity. Materials arrive by rail or truck and are processed through six work cells; all operations are remotely controlled from consoles in the control room within the building. Typical operations are pull-apart of gun ammunition, depriming of cartridge cases, collection of smokeless powder, and defusing of projectiles.

Initially, access to the energetic material is required. This is accomplished by pulling apart, sawing through, unscrewing, or drilling into the round. These operations are all preliminary to the further processing of the energetic material (e.g., by SCF extraction of the moieties).

Once access is achieved, the explosives are recovered by melt-out, steam-out, or high-pressure water wash-out. In the melt-out process, racks containing the ordnance are raised to the upper deck by an overhead crane and placed into steam autoclaves. The molten explosive is collected in kettles and poured onto a conveyor where it solidifies and is flaked into small pieces,

which are then packaged and transported to storage for sales. In the steam-out process, large items, such as mines and depth charges, are lifted to the upper deck and washed out by steam lances. The explosive flows by gravity down through a dewaterer and melt kettles and then onto a flaking conveyer, and it then is packaged for storage resale. In the wash-out process, water is used at 10,000 psi to remove pressed explosives from munition bodies. This facility has also been used to remove Explosive D and solid-rocket propellants by water-jet technology. Pressed explosives, such as Composition A3, are then put through a dryer and packaged for transport to storage for possible sale. The process water is treated and recycled.

Three furnaces are used to deactivate/decontaminate various items. They have two rotary-type furnaces and a flashing furnace. The rotary furnaces are used to process small-arms, fuse, booster, and other small-type ammunition. The refuse from the furnaces is deposited onto a second conveyor and carried to a magnetic (ferrous/nonferrous) separator where the scrap is dumped into semitrailers. In the flashing furnace, moderate-sized items (previously cleaned out in other operations) are decontaminated.

The waste water from the entire facility is treated at the Process Water Treatment Facility. It is pumped to a flotation clarifier where alum and polymer are added to expedite the coagulation process. The agglomerated particles settle to the bottom and are removed by positive displacement pumps. The sludge from the bottom is approximately 5% solids and is dewatered in a filter press. The clarified water is then passed through multimedia filters that remove any agglomerated particles that did not settle out. Next, the water flows through activated carbon columns that remove the organic contamination. Water entering the carbon columns normally has a pH of between 9 and 11, and exits in the range of 6 to 8 pH. The facility operates consistent with National Pollution Discharge Elimination System requirements and permits.

9. SUMMARY

Throughout the course of this task, many consultations and discussions were held, during which, suggestions were made and ideas exchanged, all with the aim of improving the safety of

SCF extraction of energetic moieties from explosive formulations. Check valves were added, and their location changed. Filters were added, check valves reversed, and plumbing modified. Changes in procedure and many other safety-related recommendations were also made.

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6. AUTHOR(S) Lawrence J. Vande Kieft and Warren W. Hillstrom				
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13. ABSTRACT (Maximum 200 words) <p>The Department of Defense (DOD) has a large inventory of ammunition, some of which is becoming older than desired for retention in storage. More than 400 kton are currently scheduled for demilitarization. Of this material, a significant fraction is Composition B, which is 60% RDX and 40% TNT, with 1% wax added. Research at the U.S. Army Research Laboratory (ARL) and elsewhere, particularly at the Phasex Corporation, Lawrence, MA, has demonstrated the feasibility of separating the energetic moieties by use of supercritical CO₂. No special hazards were identified, and no chemical incompatibilities were found. The system is environmentally friendly in that it is a closed-loop system, and no hazardous materials can escape. Also, this system is designed for remote operation. This report deals with potential hazards from the scale-up of the system with which the feasibility of the concept was demonstrated.</p>				
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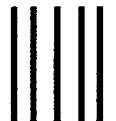
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